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by George T. Dewing, Franklyn R. Kellogg, Bradley R. Klotz, and Laszlo J. Kecskes

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14. ABSTRACT

A method for the preparation of fine powders for microscopic examination of their cross section is described. Heavily worked nickel-coated aluminum powders were mounted in epoxy and polished to a mirror finish to allow for the characterization of their interior structure. The method presented would be applicable to all types of finely divided media.

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1. Introduction

The alloying process between elemental nickel and aluminum precursors is being studied on a microscopic level. We conduct alloying by subjecting a specially prepared nickel-coated aluminum powder to extreme deformation in a high energy mechanical ball mill. The breakdown, deformation, and intimate mixing between the two elements take place very rapidly so that microscopic observation of the exterior surface of the powders is insufficient to adequately interpret the effectiveness of the milling process. Particularly, usually one element smears over the other, obscuring the mixing process. Consequently, examination of the exterior morphology must be complemented by an examination of the interior of the particles. Therefore, a need arose to develop a reliable technique to facilitate the evaluation of cross sections of a representative population of the fine particles.

2. Experimental Procedure

To demonstrate the powder preparation technique, two nickel-coated aluminum powder samples, labeled A and B, were prepared in a high energy ball mill. Two grams of each composition were weighed and added to separate steel milling vials with two 7/16-inch steel pellets and five 1/16-inch steel pellets in each vial. The amount of powder used in this experiment is more than enough for preparing microscopic specimens. On many occasions, there is much less powder available for mounting. The specimen mounting technique described offers sufficient flexibility for larger and smaller amounts of powder.

The vials containing the powder samples were loaded into the mill, which was situated in a glove box equipped with a cooling system. The glove box was then filled with argon gas and evacuated several times so that it would be purged of air. Milling was performed inside the cooled glove box in an argon atmosphere to prevent oxidation of the powders, as well as overheating of the vials. Each composition was run at nine different time intervals: the first four runs were at 5, 10, 15, and 30 minutes, and the other five runs were at 1, 2, 4, 6, and 8 hours. Additional samples of the B composition were mixed with 30 milliliters of ethanol and milled for the same time periods (four runs at 5, 10, 15, and 30 minutes and five runs at 1, 2, 4, 6, and 8 hours). After milling, the samples were removed from the vials and bagged. (The samples milled in ethanol were exposed to air and allowed to air dry in the vials before being removed.)

Representative samples of each milled powder then needed to be mounted in an epoxy resin and polished to allow for cross-sectional examination by scanning electron microscopy (SEM). The technique developed to obtain the best representation of a large population of particles for use in microscopy is shown in the schematic in figure 1. First, we created a loosely packed powder plug by pouring powder from each milled sample into the tip of a small, 1/4-inch-diameter plastic pipette. Enough powder was used to create a plug approximately 1/4-inch high from the tip of the pipette. After packing each pipette, we added a few drops of mounting resin to each sample. The sample was placed in a bell jar, and a partial vacuum (about 1 torr) was "pulled" to assist in drawing the liquid resin into the powder bed. If the application of a vacuum was not adequate to draw the resin into the powder bed, a thin wooden pick was used to agitate the mixture to properly disperse the powder in the epoxy. The samples were then allowed to cure overnight. After curing, the plastic pipette sheaths were removed and the hardened pre-mounted samples were placed into 1-1/4-inch-diameter plastic cups with mounting clips to hold the samples vertically in the bottom of the cup. Enough mounting resin was then added to the cups to create approximately 3/4-inch-thick standard SEM mounts. The samples were then allowed to cure under vacuum.

After mounting, the samples were polished for microscopy. They were sequentially ground on a series of silicon carbide papers, with grit sizes of 320, 500, 1000, 1200, 2400, and 4000 (increasing grit number corresponds to a decreasing abrasive size). Samples were ground for 20 to 30 seconds on each paper. Final polishing was done on a 1-micron diamond wheel for 20 to 30 seconds. After the mounts were cleaned in alcohol to remove dirt, polishing residue, and other contaminants, they were examined by SEM.

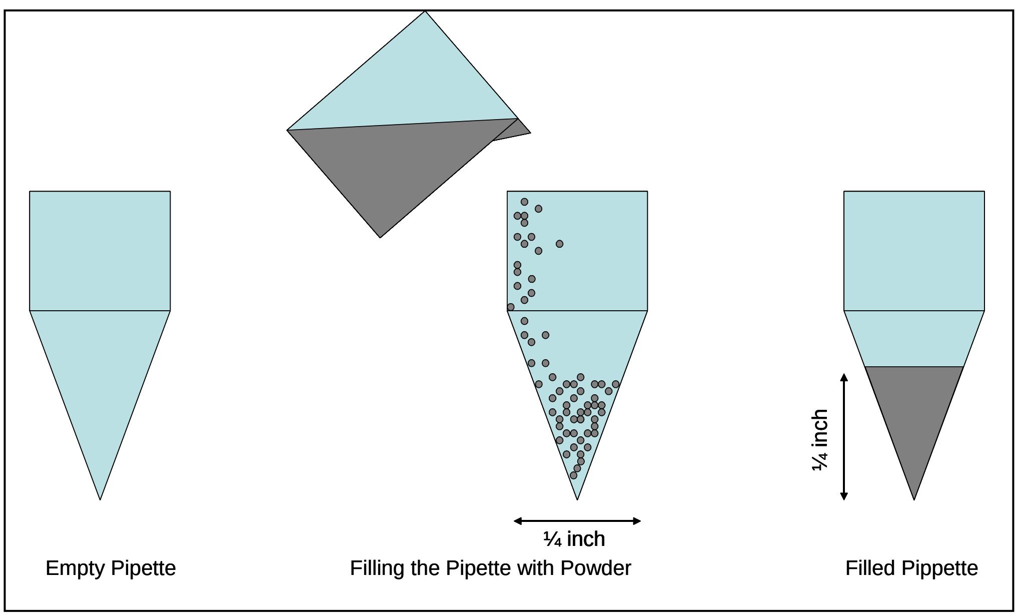

Figure 1. Schematic of preparation of the powder plug which is filled with resin, allowed to cure, then remounted and prepared metallographically for microscopy.

3. Results and Discussion

The use of the mounting technique described previously is critical to generate a large enough collection of particles for examination by SEM. Previous attempts at mounting powders for cross-sectional examination involved pouring the powder directly into the 1-1/4-inch diameter mounting cup and adding the epoxy resin. However, this technique resulted in a thinly spread dispersion of powder in the epoxy mount, which was often polished away during grinding of the sample. It also made the creation of the conductive path required for SEM examination difficult in that the powder was too spread out to adequately create a path between the particles.

SEM micrographs of the composition-A powder at different milling times are shown in figure 2, which displays the rapid breakdown and comminution (pulverization) of the coated powder. In the images, the light areas correspond to the nickel coating, the dark gray areas are the aluminum particles, and the black regions correspond to the epoxy resin. The nickel layer on the particles was observed to separate and fragment, forming alternating layers of nickel and aluminum that continued to be refined and more intimately mixed. Samples of composition A milled for longer times and samples of composition B have been prepared but not yet examined in the SEM.

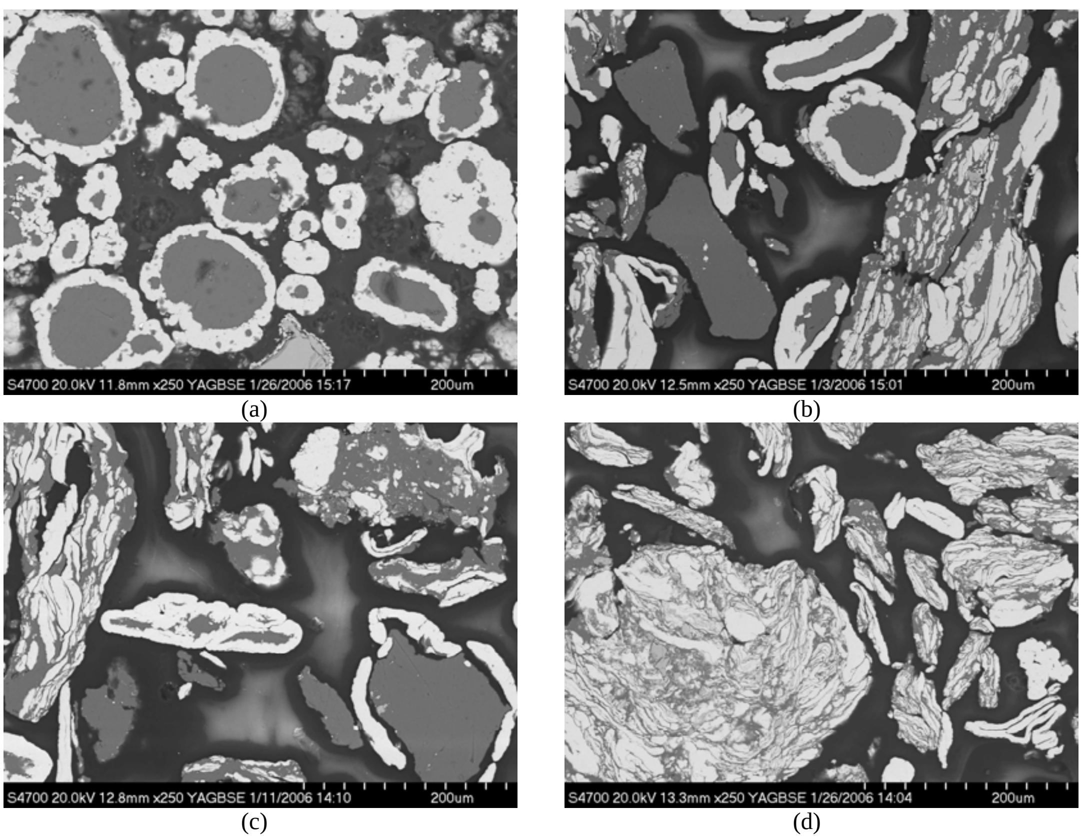

Figure 2. Backscattered SEM micrographs of the high energy milled nickel-coated aluminum in the (a) as-received, (b) 5-minute milled, (c) 10-minute milled, and (d) 30-minute milled conditions demonstrate the rapid intermixing of the two precursors.

The micrographs clearly illustrate that this modification in the mounting technique permits the simultaneous evaluation of several particles. During pre-mounting and re-mounting, the epoxy appeared to infiltrate well into the powder plug so that there are no apparent voids. Also, as apparent in the figures, there is little or no debonding or pull-out[1] of the nickel layer. This is indicative that there was good adhesion between the epoxy and powder particles. While this mounting technique did require the additional step of creating the powder plug, the overall procedure was seen to expedite the microscopic evaluation process, as well as improve the particle adhesion and retention in the mounting medium.

4. Conclusions

A convenient technique was developed for the mounting and polishing of loose powders. This technique permits the examination of finely divided powder particle cross sections. While the overall research effort is continuing, it is of interest to observe the deformation and extent of intermixing between the nickel and aluminum precursors after milling.

[1]Pull-out refers to the particles separating from the epoxy mount, leaving a hole behind.

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